PCT/EP2004/051235

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Patentanmeldung Nr. Patent application No. Demande de brevet n°

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Der Präsident des Europäischen Patentamts;
Im Auftrag

For the President of the European Patent Office

Le Président de l'Office européen des brevets
p.o.

R C van Dijk

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(Falls die Bezeichnung der Erfindung nicht angegeben ist, siehe Beschreibung.
If no title is shown please refer to the description.
Si aucun titre n'est indiqué se referer à la description.)

Polyorganosiloxanes

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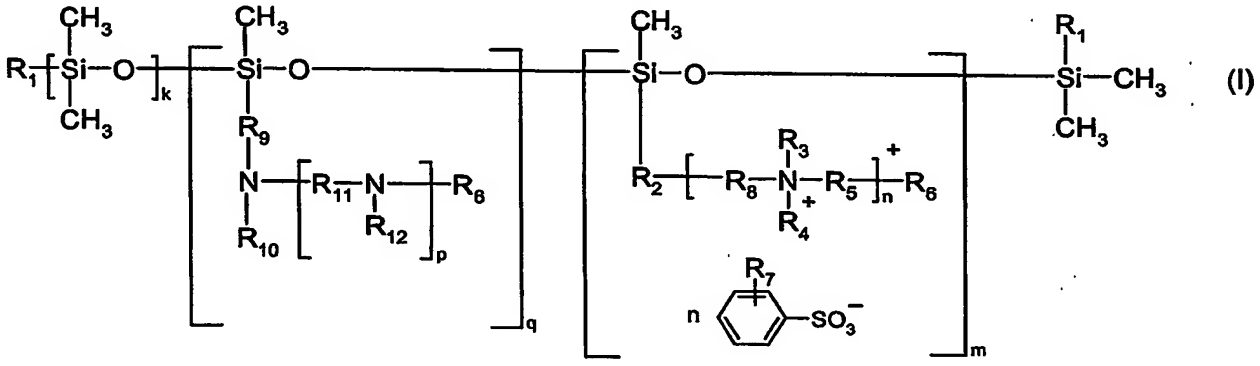
Polyorganosiloxanes

The invention relates to polyorganosiloxanes having a concentration of nitrogen of > 0.8 % by weight (wt-%), based on the total weight of the polyorganosiloxane, to compositions comprising such polyorganosiloxanes and to the treatment of fiber materials with such compositions.

In DE 19652524A1, compositions comprising polyorganosiloxanes having a concentration of nitrogen in the polyorganosiloxane of 0.21 and 0.8 wt-%, based on the total amount of polyorganosiloxane, are explicitly disclosed.

The known polyorganosiloxane do still show some disadvantages. Therefore, the goal of the present patent application was to provide polyorganosiloxanes with improved properties. Such properties are i.e. the recovery angle of the treated fabric, hydrophilicity, antistatic and softness of the treated fabric.

The invention relates to polyorganosiloxanes having the following formula (I)



(I)

in which said structural units may be distributed over the polysiloxane chain in any order, in which

each R_1 is independently from each other –OH; -OC_1-C_8alkyl or -CH_3,

R_2 is a linear or branched C_1-C_{16}alkylene,

R_3 and R_4 are independently from each other linear C_1-C_8alkyl; branched or cyclic C_3-C_8alkyl;

R_5 and R_8 are independently from each other linear or branched C_1-C_{16}alkylene,

R_6 and R_7 are independently from each other H; linear C_1-C_8alkyl; branched or cyclic C_3-C_8alkyl,

R_9 is a linear or branched C_1-C_{16}alkylene,
R_{10} and R_{12} are independently from each other H; linear C_1-C_8alkyl; branched or cyclic C_3-C_8alkyl,
R_{11} is a linear or branched C_1-C_{16}alkylene,
n is 1, 2 or 3,
p is 0, 1 or 2,
the sum of k, m and q is 25 to 900,
whereby the concentration of nitrogen in the polyorganosiloxane is > 0.8 wt-%, based on the total weight of the polyorganosiloxane.

Linear C_1-C_8alkyl can be methyl, ethyl, propyl, butyl, propyl, pentyl, hexyl or octyl.

Branched C_3-C_8alkyl can be any possible isomer of linear C_3-C_8alkyl. Examples are – $CH(CH_3)_2$, $-CH(CH_3)CH_2CH_3$, $-(CH_2)_{1-5}CH(CH_3)_2$, $-C(CH_3)_3$, , $-(CH_2)_{1-4}CH(CH_3)CH_2CH_3$, - $CH(CH_3)$ $(CH_2)_{1-6}CH_3$, $-CH(CH_3)(CH_2)_{1-3}CH(CH_3)_2$, $C(CH_3)_2(CH_2)_{1-4}CH_3$, $-(CH_2)_{1-4}C(CH_3)_3$.

Cyclic C_3–C_8alkyl can be cyclopropyl, cyclobutyl, cyclopentyl, cyclohexyl, cycloheptyl or cyclooctyl. Preferably cyclopentyl, cyclohexyl, cycloheptyl or cyclooctyl as well as alkyl substituted C_4-C_7cylcoalkyl moieties.

Linear or branched C_1 – C_{16} alkylene can be methylen, ethylene, propylene, butylene, pentylene, hexylene, heptylene, octylene, nonylene, decanylene, undecanylene, dodecanylene, tridecanylene, tetradecanylene, pentadecanylene and hexadecanylene as well as all possible branched isomers thereof.

Preferably, R_2 is a linear or branched C_1-C_{12}alkylene, more preferably C_1-C_8alkylene, especially preferred C_1-C_4alkylene.
Preferably, R_3 and R_4 are independently from each other linear or branched C_1-C_6alkyl or cyclic C_4-C_8 alkyl, more preferably linear or branched C_1-C_4alkyl or cyclopentyl, cyclohexyl or cyclooctyl.
Preferably, R_5 and R_8 are independently from each other linear or branched C_1-C_{12}alkylene, more preferably C_1-C_8alkylene, especially preferred C_1-C_4alkylene.

Preferably, R_6 and R_7 are independently from each other H; linear or branched C_1-C_6alkyl or cyclic C_4-C_8 alkyl, more preferably H; linear or branched C_1-C_4alkyl or cyclopentyl, cyclohexyl or cyclooctyl.

Preferably, R_9 is linear or branched C_1-C_{12}alkylene, more preferably C_1-C_8alkylene, especially preferred C_1-C_4alkylene.

Preferably, R_{10} and R_{12} are independently from each H; linear or branched C_1-C_6alkyl or cyclic C_4-C_8 alkyl, more preferably H; linear or branched C_1-C_4alkyl or cyclopentyl, cyclohexyl or cyclooctyl.

Preferably, R_{11} is linear or branched C_1-C_{12}alkylene, more preferably C_1-C_8alkylene, especially preferred C_1-C_4alkylene.

Preferably, n is 1, 2 or 3,

Preferably, p is 0, 1 or 2,

Preferably, the sum of k, m and q is 25 to 700, more preferably the sum of k, m and p is 25 to 500.

Preferably, the polyorganosiloxanes of the present invention have a concentration of nitrogen of ≥ 1 wt-%, more preferably of ≥ 1.5 wt-%, especially preferred of ≥ 1.5 wt-% and < 8 wt-%, very especially preferred of ≥ 1.5 wt-% and < 5 wt-%, always based on the total weight of the polyorganosiloxane.

A further embodiment of the present invention is a composition comprising the above defined polyorganosiloxanes.

Such compositions are preferably aqueous solutions or dispersions, which preferably contain from 2 wt-% to 60 wt-%of the polyorganosiloxane as defined by the invention. Depending on the chemical nature of the polysiloxane it is possible that the latter is soluble or self-dispersible in water. In the other cases, highly stable aqueous dispersions can be obtained by adding one or several dispersing agents. Suitable as dispersants are surface-active compounds known to the expert in the field of silicone emulsions. Non-ionogenic products such as fatty alcohol ethoxylates, fatty acid ethoxylates, or ethoxylated fatty amines, or cation-active dispersants such as, for example quaternized ammonium salts have to be mentioned here in particular. The amount of dispersant is in the range of, for example from 2 wt-% to 10 wt-% based on the total dispersion. The dispersions can be produced by generally known methods employed for dispersing polysiloxanes.

Polyorganosiloxanes as defined by the invention in the form of aqueous dispersions or solutions are excellently suitable for treating fiber materials, in particular flat textile structures within the framework of textile finishing or dressing. For such purposes, the solutions or dispersions may contain also other products known in the field of textile dressing such as, for example polymers with perfluoroalkyl groups for achieving oil-repelling properties; fatty acid alkanolamides; waxes in the dispersed form, or other polyorganosiloxanes. The aqueous solutions or dispersions can be applied to the fiber materials, and further processing can be carried out by generally known methods. Such aqueous solutions or dispersions are preferably applied by means of a padding process. The fiber materials are preferably flat textile structures in the form of woven or knitted fabrics, which may consist of cellulose, in particular cotton, synthetic polymers, or mixtures of said fibers.

Polyorganosiloxanes as defined by the invention are flowable. This means that they are either liquid or at least flowable at room temperature and thus do not have a solid or pasty consistency.

The compositions according to the present invention comprise from 2 to 60 wt-%, preferably from 5 – 50 wt-%, more preferably from 10 – 40 wt-%, of the polyorganosiloxanes based on the total weight of the composition.

The polyorganosiloxanes according to formula (I) as well as their production are known. The composition can be produced in analogy to known processes. Such a process is for example disclosed in DE 19652524A1.

The composition according to the present invention may further comprise buffers; hydrotropica, such as polyfunctional alcohol, i.e. 1,2-propylenglycol or dipropylenglycol; organic or anorganic acid, such as formic acid, acetic acid, glycol adic, oxalic acid, citric acid, citric acid, lactic acid, hydrochloric acid, sulfuric acid or phosphoric acid; and further common auxilaries depending on the use.

A further embodiment of the present invention are softnener compositions comprising

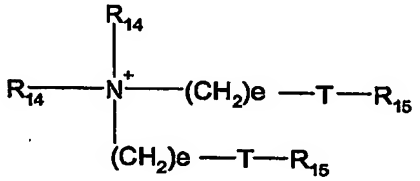
a) a fabric softener; and
b) an aqueous dispersion as defined above.

Fabric softeners, especially hydrocarbon fabric softeners, suitable for use herein are selected from the following classes of compounds:

(i) Cationic quaternary ammonium salts. The counter ion of such cationic quaternary ammonium salts may be a halide, such as chloride or bromide, methyl sulphate, or other ions well known in the literature. Preferably the counter ion is methyl sulfate or any alkyl sulfate or any halide, methyl sulfate being most preferred for the dryer-added articles of the invention.

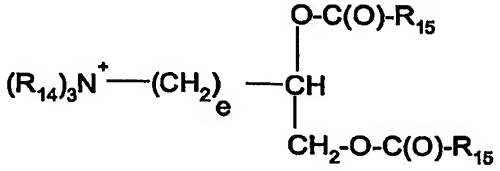
Examples of cationic quaternary ammonium salts include but are not limited to:

(1) Acyclic quaternary ammonium salts having at least two C_8 to C_{30}, preferably C_{12} to C_{22} alkyl or alkenyl chains, such as: ditallowdimethyl ammonium methylsulfate, di(hydrogenated tallow)dimethyl ammonium methylsulfate, di(hydrogenated tallow)dimethyl ammonium methylchloride, distearyldimethyl ammonium methyl-sulfate, dicocodimethyl ammonium methylsulfate and the like. It is especially preferred if the fabric softening compound is a water insoluble quaternary ammonium material which comprises a compound having two C_{12} to C_{18} alkyl or alkenyl groups connected to the molecule via at least one ester link. It is more preferred if the quaternary ammonium material has two ester links present. An especially preferred ester-linked quaternary ammonium material for use in the invention can be represented by the formula:



wherein each R_{14} group is independently selected from C_1 to C_4 alkyl, hydroxyalkyl or C_2 to C_4 alkenyl groups; T is either -O-C(O)- or -C(O)-O-, and wherein each R_{15} group is independently selected from C_8 to C_{28} alkyl or alkenyl groups; and e is an integer from 0 to 5.

A second preferred type of quaternary ammonium material can be represented by the formula:



wherein R_{14}, e and R_{15} are as defined above.

(2) Cyclic quaternary ammonium salts of the imidazolinium type such as di(hydrogenated tallow)dimethyl imidazolinium methylsulfate, 1-ethylene-bis(2-tallow-1-methyl) imidazolinium methylsulfate and the like;

(3) Diamido quaternary ammonium salts such as: methyl-bis(hydrogenated tallow amidoethyl)-2-hydroxethyl ammonium methyl sulfate, methyl bi(tallowamidoethyl)-2-hydroxypropyl ammonium methylsulfate and the like;

(4) Biodegradable quaternary ammonium salts such as N,N-di(tallowoyl-oxy-ethyl)-N,N-dimethyl ammonium methyl sulfate and N,N-di(tallowoyl-oxy-propyl)-N,N-dimethyl ammonium methyl sulfate. Biodegradable quaternary ammonium salts are described, for example, in U.S. Patents 4,137,180, 4,767,547 and 4,789,491 incorporated by reference herein.

Preferred biodegradable quaternary ammonium salts include the biodegradable cationic diester compounds as described in U.S. Patent 4,137,180, herein incorporated by reference.

(ii) Tertiary fatty amines having at least one and preferably two C_8 to C_{30}, preferably C_{12} to C_{22} alkyl chains. Examples include hardened tallow-di-methylamine and cyclic amines such as 1-(hydrogenated tallow)amidoethyl-2-(hydrogenated tallow) imidazoline. Cyclic amines, which may be employed for the compositions herein, are described in U.S. Patent 4,806,255 incorporated by reference herein.

(iii) Carboxylic acids having 8 to 30 carbons atoms and one carboxylic group per molecule. The alkyl portion has 8 to 30, preferably 12 to 22 carbon atoms. The alkyl portion may be linear or branched, saturated or unsaturated, with linear saturated alkyl preferred. Stearic acid is a preferred fatty acid for use in the composition herein. Examples of these carboxylic acids are commercial grades of stearic acid and palmitic acid, and mixtures thereof, which may contain small amounts of other acids.

(iv) Esters of polyhydric alcohols such as sorbitan esters or glycerol stearate. Sorbitan esters are the condensation products of sorbitol or iso-sorbitol with fatty acids such as stearic acid.

Preferred sorbitan esters are monoalkyl. A common example of sorbitan ester is SPAN® 60 (ICI) which is a mixture of sorbitan and isosorbide stearates.

(v) Fatty alcohols, ethoxylated fatty alcohols, alkyphenols, ethoxylated alkyphenols, ethoxylated fatty amines, ethoxylated monoglycerides and ethoxylated diglycerides.

(vi) Mineral oils, and polyols such as polyethylene glycol.

These softeners are more definitively described in U.S. Patent 4,134,838 the disclosure of which is incorporated by reference herein. Preferred fabric softeners for use herein are acyclic quaternary ammonium salts. Mixtures of the above mentioned fabric softeners may also be used.

The fabric softening composition employed in the present invention preferably contains about 0.1 to about 95 wt-%, based on the total weight of the fabric softening composition, of the fabric softening component. Preferred is an amount of 0.5 to 50 wt-%, especially an amount of 2 to 50 wt-% and most preferably an amount of 2 to 30 wt-%.

The amount of the polyorganosiloxanes in the fabric softening composition is preferably from 0.01 to 50 wt-%, based on the total weight of the fabric softening composition. Preferred is an amount of 0.01 to 30 wt-%, especially an amount of 0.05 to 30 wt-% and most preferably an amount of 0.05 to 18 wt-%.

The fabric softening composition may also comprise additives which are customary for standard commercial fabric softening compositions, for example alcohols, such as ethanol, n-propanol, i-propanol, polyhydric alcohols, for example glycerol and propylene glycol; amphoteric and nonionic surfactants, for example carboxyl derivatives of imidazole, oxyethylated fatty alcohols, hydrogenated and ethoxylated castor oil, alkyl polyglycosides, for example decyl polyglucose and dodecylpolyglucose, fatty alcohols, fatty acid esters, fatty acids, ethoxylated fatty acid glycerides or fatty acid partial glycerides; also inorganic or organic salts, for example water-soluble potassium, sodium or magnesium salts, non-aqueous solvents, pH buffers, perfumes, dyes, hydrotropic agents, antifoams, anti redeposition agents, enzymes, optical brighteners, antishrink agents, stain removers, germicides, fungicides, dye fixing agents or dye transfer inhibitors (as described in WO-A-

02/02865), antioxidants, corrosion inhibitors, wrinkle recovery or wet soiling reduction agent, such as polyorganosiloxanes. The latter two additives are described in WO0125385.

Such additives are preferably used in an amount of 0 to 30 wt-%, based on the total weight of the fabric softening composition. Preferred is an amount of 0 to 20 wt-%, especially an amount of 0 to 10 wt-% and most preferably an amount of 0 to 5 wt-%, based on the total weight of the fabric softening composition.

The fabric softener compositions are preferably in liquid aqueous form. The fabric softener compositions preferably contain a water content of 25 to 90 wt-%, based on the total weight of the composition. More preferably the water content is 50 to 90 wt-%, especially 60 to 90 wt-%.

The fabric softener compositions preferably have a pH value from 2.0 to 9.0, especially 2.0 to 5.0.

The fabric softener compositions can, for example, be prepared as follows:
Firstly, an aqueous formulation of the cationic polymer is prepared as described above. The fabric softener composition according to the invention is usually, but not exclusively, prepared by firstly stirring the active substance, i.e. the hydrocarbon based fabric softening component, in the molten state into water, then, where required, adding further desired additives and, finally, adding the formulation of the cationic polymer. The fabric softener composition can, for example, also be prepared by mixing a preformulated fabric softener with the cationic polymer.

These fabric softener compositions are traditionally prepared as dispersions containing for example up to 30 wt-% of active material in water. They usually have a turbid appearance. However, alternative formulations usually containing actives at levels of 5 to 40 wt-% along with solvents can be prepared as microemulsions, which have a clear appearance (as to the solvents and the formulations see for example US-A-5,543,067 und WO-A-98/17757).

Examples of suitable textile fibre materials which can be treated with the liquid rinse conditioner composition are materials made of silk, wool, polyamide, acrylics or polyurethanes, and, in particular, cellulosic fibre materials of all types. Such fibre materials

are, for example, natural cellulose fibres, such as cotton, linen, jute and hemp, and regenerated cellulose. Preference is given to textile fibre materials made of cotton. The fabric softener compositions are also suitable for hydroxyl-containing fibres which are present in mixed fabrics, for example mixtures of cotton with polyester fibres or polyamide fibres. The recovery angle of the textile treated with these compositions are improved.

Referring to the following Examples, given by way of illustration, will have a better understanding of the present invention and of its many advantages. The percentages given in the examples are percentages by weight.

Example 1 (preparation of the rinse conditioners)

The liquid rinse conditioners are prepared by using the procedure described below. This type of fabric rinse conditioners is normally known under the name of "triple strength" or "triple fold" formula.

75 g of water is heated to 40°C. 15 g of the molten fabric softener Di-(nortallow carboxy-ethyl-)hydroxyethyl-methylammonium-methosulfate (Rewoquat® WE 18 available from Witco) is added to the heated water under stirring and the mixture is stirred for 1 hour at 40°C. Afterwards the aqueous softener solution is cooled down to below 30°C while stirring. When the solution cools down 0.1 g of magnesium chloride is added and the pH is adjusted to 3.2 with 0.1 N hydrochloric acid. The formulation is then filled up with water to 100 g.

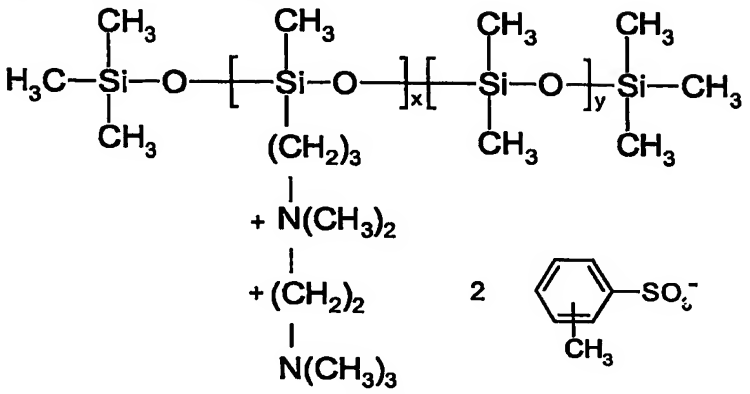
The rinse conditioner formulation as described above was used as a base formulation. In a final step the fabric softener is mixed with a separately prepared "PDMS"-Formulation. The fabric softener formulations used in the following examples are listed in the following Table 1.

Table 1 (rinse conditioner formulations used in the application test for 1 kg wash load)

Rinse conditioner formulation	"PDMS"-Formulation (calculated on solid content of the formulation)	Fabric softener Base Formulation	pH
A	0.28 g of Type I	14 g	3.2
B	0.28 g of Type II	14 g	3.2
C	0.28 g of Type III	14 g	3.2
D	0.28 g of Type IV	14 g	3.2

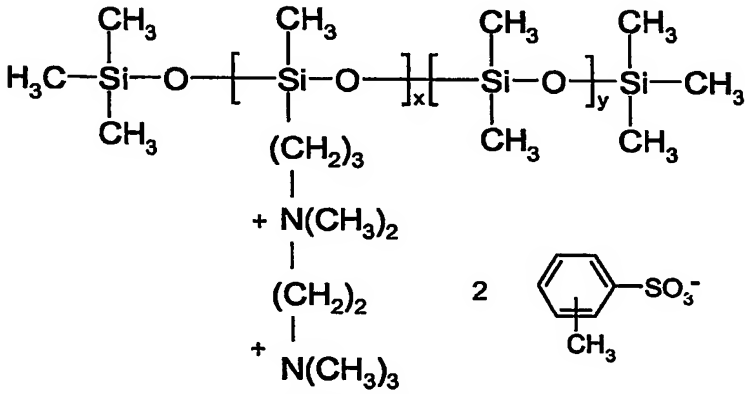
Types of "PDMS"-Formulation used

Type I (State of the art):



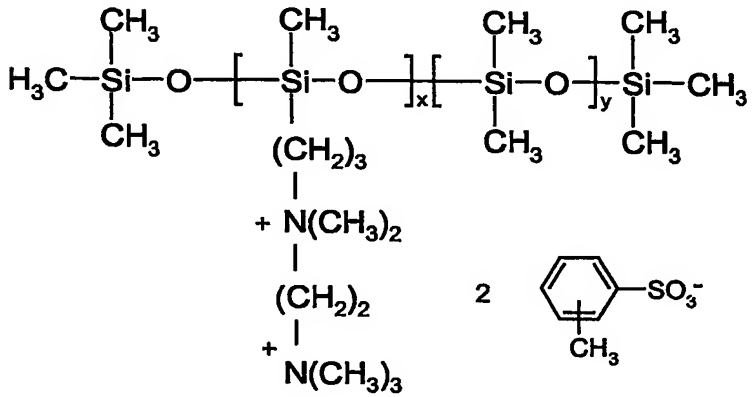
x:y = 1:50 and the content of nitrogen 0.7%.

Type II:



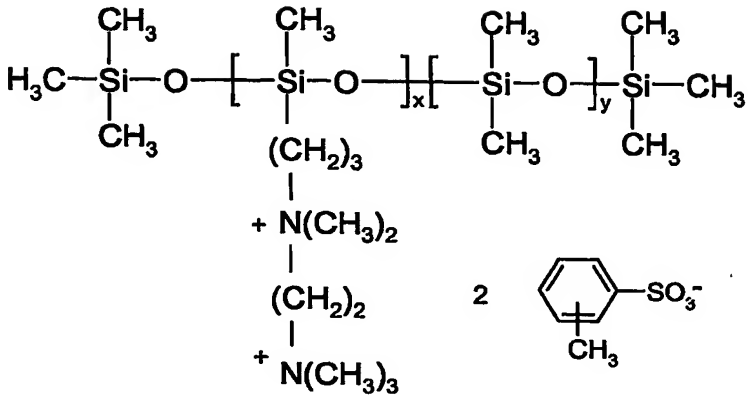
x:y = 1:23 and the content of nitrogen 1.7%.

Type III:



x:y = 1:10 and the content of nitrogen 3.0%.

Type IV:



x:y = 1:3 and the content of nitrogen 7.1%.

Example 2 (Improvement of crease recovery angle)

The formulated rinse conditioners (see Table 1) are applied according to the following procedure:

Woven cotton (without textile finishing, 120 g/m^2) swatches of size of 34 cm by 34 cm are washed together with ballast material (cotton and cotton/polyester) in an AEG Oeko Lavamat 73729 washing machine maintaining the washing temperature at 40°C. The total fabric load of 1 kg is washed for 15 minutes with 33 g of ECE Color Fastness Test Detergent 77 (Formulation January 1977, according to ISO 105-CO6). The rinse conditioner formulation as described in Table 1 is applied in the last rinse cycle at 20°C. After rinsing with the formulation the textile swatches are dried on a washing line at ambient temperature.

Evaluation of crease recovery angle

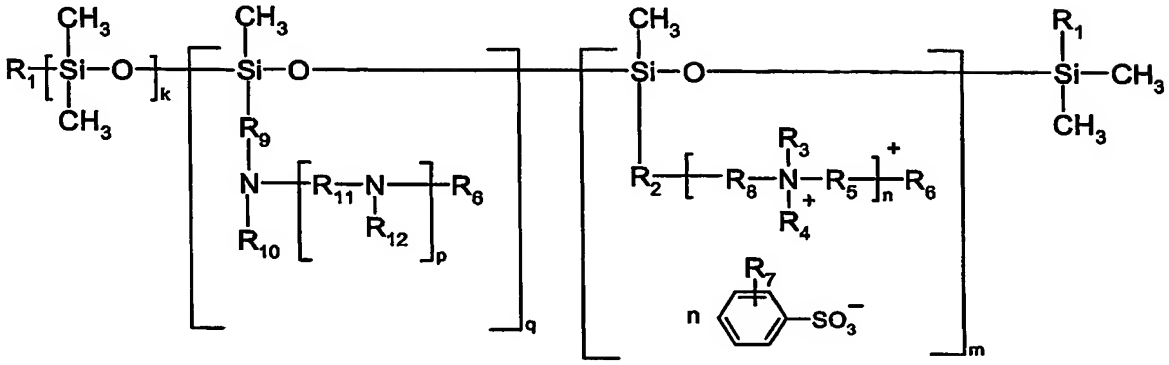
Table 2 (Results of the evaluation of crease recovery angle on cotton/polyester by DIN 53890 method, 1 kg loading, 30 minutes relaxing)

Sample of rinse conditioner formulation	Crease recovery angle(mean value of warp and weft)
A (Prior Art)	63
B	71
C	71
D	70

The above results show a marked improvement in crease recovery angle of sample B to D for the textile fabric material treated with compositions of the present invention.

CLAIMS

1. A polyorganosiloxane having the following formula (I)



(I)

in which said structural units may be distributed over the polysiloxane chain in any order, in which

each R_1 is independently from each other –OH; -OC_1-C_8alkyl or -CH_3,

R_2 is a linear or branched C_1-C_{16}alkylene,

R_3 and R_4 are independently from each other linear C_1-C_8alkyl; branched or cyclic C_3-C_8alkyl;

R_5 and R_8 are independently from each other linear or branched C_1-C_{16}alkylene,

R_6 and R_7 are independently from each other H; linear C_1-C_8alkyl; branched or cyclic C_3-C_8alkyl,

R_9 is a linear or branched C_1-C_{16}alkylene,

R_{10} and R_{12} are independently from each other H; linear C_1-C_8alkyl; branched or cyclic C_3-C_8alkyl,

R_{11} is a linear or branched C_1-C_{16}alkylene,

n is 1, 2 or 3,

p is 0, 1 or 2,

the sum of k, m and q is 25 to 900,

whereby the concentration of nitrogen in the polyorganosiloxane is > 0.8 wt-%, based on the total weight of the polyorganosiloxane.

2. A polyorganosiloxane according to Claim 1 wherein

R_2 is a linear or branched C_1-C_{12}alkylene;

R_3 and R_4 are independently from each other linear or branched C_1-C_6alkyl or cyclic C_4-C_8 alkyl;

R_5 and R_8 are independently from each other linear or branched C_1-C_{12}alkylene;
R_6 and R_7 are independently from each other H; linear or branched C_1-C_6alkyl or cyclic C_4-C_8 alkyl;
R_9 is a linear or branched C_1-C_{12}alkylene;
Preferably R_{10} and R_{12} are independently from each other H; linear or branched C_1-C_6alkyl or cyclic C_4-C_8alkyl; and
R_{11} is a linear or branched C_1-C_{12}alkylene.

3. A polyorganosiloxane according to Claims 1 or 2 wherein the concentration of nitrogen is ≥ 1 wt-%, based on the total weight of the polyorganosiloxane.

4. A polyorganosiloxane according to Claims 1 or 2 wherein the concentration of nitrogen is ≥ 1.5 wt-%, based on the total weight of the polyorganosiloxane.

5. A polyorganosiloxane according to Claims 1 or 2 wherein the concentration of nitrogen is ≥ 1.5 wt-% and < 8 wt-%, based on the total weight of the polyorganosiloxane.

6. A polyorganosiloxane according to Claims 1 or 2 wherein the concentration of nitrogen is ≥ 1.5 wt-% and < 5 wt-%, based on the total weight of the polyorganosiloxane.

7. A polyorganosiloxane according to any one of the preceeding wherein the sum of k, m and q is 25 to 700, preferably 25 to 500.

8. A composition according comprising at least one polyorganosiloxane as defined in Claims 1 - 7.

9. A composition according comprising to Claim 8 comprising from 2 wt-% to 60 wt-%, based on the total weigth of the composition of at least one polyorganosiloxane.

10. A composition according to Claim 8 or 9 comprising a fabric softener.

11. A composition according to Claim 10 comprising about 0.1 to about 95 wt-%, based on the total weight of the composition, of the fabric softening component.

12. A composition according to Claims 8 or Claim 9 comprising 0 to 30 wt-%, based on the total weight of the composition, additives which are customary for standard commercial fabric softening compositions.

13. A composition according to Claims 8 to 12 comprising 25 to 90 wt-%, based on the total weight of the composition, water.

14. A composition according to Claims 8 to 13 characterized in that the pH-value is from 2.0 to 9.0.

15. Use of a composition according to Claims 8 to 14 for the treatment of textile.

Abstract

The invention relates to polyorganosiloxanes having a concentration of nitrogen of > 0.8 % by weight (wt-%), based on the total weight of the polyorganosiloxane, to compositions comprising such polyorganosiloxanes and to the treatment of fiber materials with such compositions.


PCT/EP2004/051235